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Iododurene

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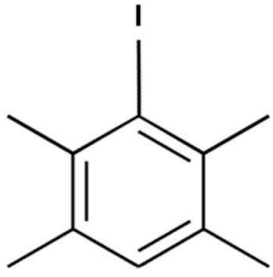
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; R factor = 0.035; wR factor = 0.039; data-to-parameter ratio = 32.4.

The title compound (systematic name: 1-iodo-2,3,5,6-tetramethylbenzene), $C_{10}H_{13}I$, crystallizes in the chiral space group $P2_12_12_1$. The I atom is displaced by 0.1003 (5) Å from the mean plane of the ten C atoms [maximum deviation = 0.018 (6) Å]. In the crystal, there are no significant intermolecular interactions present.

Related literature

For the crystal structure of bromodurene, see: Charbonneau *et al.* (1964, 1965). For the physical properties of monohalogenated derivatives of durene, see: Balcou *et al.* (1965). For standard bond lengths in similar compounds, see: Allen (2002); Hope *et al.* (1970).



Experimental

Crystal data

$C_{10}H_{13}I$
M_r = 260.11
Orthorhombic, $P2_12_12_1$
a = 5.5099 (3) Å
b = 11.8839 (5) Å
c = 15.1704 (6) Å
V = 993.34 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.16 mm^{-1}
T = 293 K
0.10 × 0.05 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.140, T_{max} = 0.193
26027 measured reflections
2271 independent reflections
2036 reflections with $I > 3\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.039
S = 1.04
3272 reflections
101 parameters
36 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}
Absolute structure: Flack (1983), 925 Friedal pairs
Flack parameter: −0.03 (4)

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DIRAX/LSQ* (Duisenberg *et al.*, 2003); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996) and *DIAMOND* (Brandenburg, 2012); software used to prepare material for publication: *CRYSTALS*.

The authors thank the Centre de Diffractométrie de l'Université de Rennes 1 for the opportunity to collect data on the Nonius Kappa CCD X-ray diffractometer. We would also like to thank Dr Olivier Jeannin for the useful advice he provided.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2521).

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supplementary materials

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Iododurene

Noudjoud Hamdouni, Ouarda Brihi, Mohamed Larbi Medjroubi, Jean Meinnel and Ali Boudjada

Comment

Substituted benzene compounds are generally liquid at room temperature so relatively few crystal structure analyses of such compounds have been reported. We have synthesized three mono-halogenated products of duren; chloro-, bromo- and iododurene. These derivatives are solid at 293 K and their dielectric behavior depends on the nature of the substituent, and in particular of its mass and volume. Contrary to chlorodurene the title iododurene in the solid phase has a quite low permeability equal to only 2.6, which changes abruptly at fusion to a value of ca. 4 (Balcou *et al.*, 1965). We report herein on its crystal structure.

The molecular structure of the title compound is illustrated in Fig. 1. The molecule is almost planar with the I atom displaced from the mean plane of the nine C atoms [maximum deviation 0.018 (6) Å for the methyl C atoms C7 and C9] by 0.1003 (5) Å. The average endocyclic angles facing the I atom and the methyl groups are 124.98 (10)° and 117.41 (10)°, respectively. The structural study did not reveal any disorder and the average intramolecular bond lengths, [C_{ar}-I = 2.139 (3) Å, C_{ar}-C_{ar} = 1.400 (5) Å and C_{ar}-C_{Me} = 1.498 (6) Å], as well as the average single C_{ar}-H bond length [0.940 Å], agree with the distances reported in the literature [Cambridge Structural Database (Allen, 2002); Hope *et al.*, 1970].

In the crystal, the *a* axis is very short, so the molecules stack along this direction (Fig. 2). The best mean plane passing through the carbon and iodine atoms, calculated with the program Molax of *CRYSTALS* (Betteridge *et al.*, 2003), indicated that the angle between the normal to this mean plane is ca. 50.1° relative to the *a* axis, ca. 44.8° relative to the *b* axis and ca. 74.5° relative to the *c* axis (Fig. 3).

The crystals obtained sublimate a little at ambient temperature. Moreover, at room temperature iododurene and the isotypic product bromodurene (Charbonneau *et al.*, 1964, 1965) crystallize in the same orthorhombic space group with fixed orientations of the dipolar grouping not presenting dielectric dispersion. This fact could be interpreted by the importance of the distribution of the masses of the substituents, that inhibit the possibility of molecular reorientation like in chlorodurene (Balcou *et al.*, 1965).

Experimental

The title compound was synthesized by direct iodination of durene. Equimolecular quantities of iodine and durene were dissolved in pure acetic acid at 343 K. Progressive iodination was obtained by oxidation of durene using drops of pure sulfuric and nitric acids diluted in acetic acid. Total precipitation of iododurene was obtained by dilution with water. Purification was done by chromatography on a silica column and finally precipitation of a solution in chloroform, giving colouress needle-like crystals.

Refinement

All H atoms were localized in a difference Fourier map. The C-bound H atoms were included in calculated positions and treated as riding atoms: C-H = 0.94 Å for CH H atoms, and 0.95 - 0.97 Å for CH_3 H atoms, with $U_{iso}(H) = k \times U_{eq}(C)$ where k = 1.5 for CH_3 H atoms and = 1.2 for other H atoms.

Computing details

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DIRAX/LSQ* (Duisenberg *et al.*, 2003); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996) and *DIAMOND* (Brandenburg, 2012); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

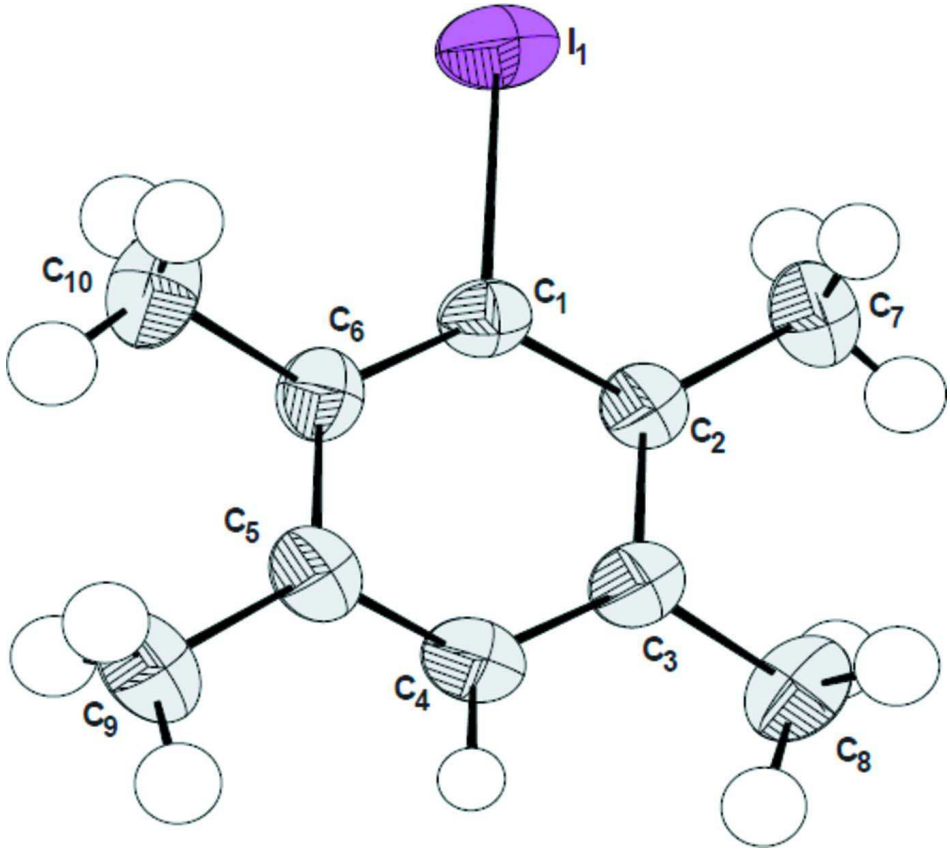

Figure 1

The molecular structure of the title molecule, with atom numbering. The displacement ellipsoids are drawn at the 50% probability level.

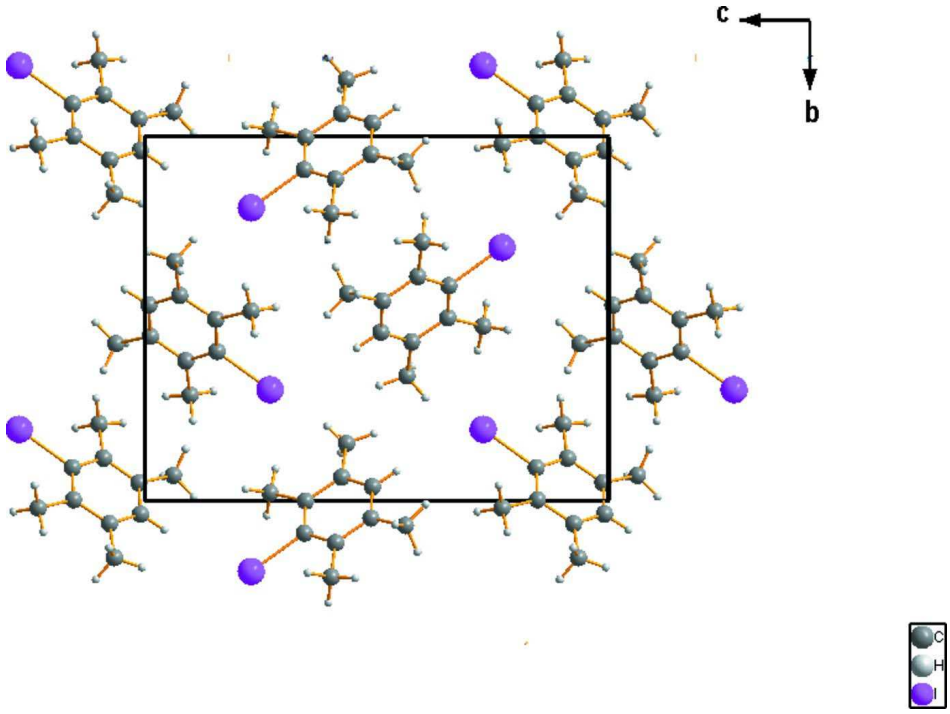

Figure 2

A view along the *a* axis of the crystal packing of the title compound.

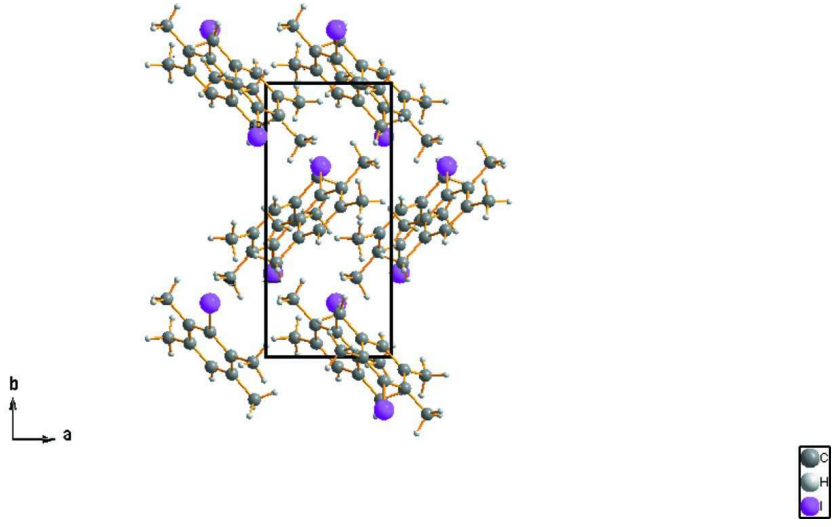

Figure 3

A view along the c axis of the crystal packing of the title compound.

1-Iodo-2,3,5,6-tetramethylbenzene

Crystal data

$C_{10}H_{13}I$
$M_r = 260.11$
Orthorhombic, $P2_12_12_1$
$a = 5.5099$ (3) Å
$b = 11.8839$ (5) Å
$c = 15.1704$ (6) Å
$V = 993.34$ (8) Å^3
$Z = 4$

$F(000)$ = 503.986
D_x = 1.739 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8232 reflections
θ = 3.7–27.5°
μ = 3.16 mm^{-1}
T = 293 K
Needle, colourless
0.10 × 0.05 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
CCD scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.140, T_{max} = 0.193
26027 measured reflections
2271 independent reflections
2036 reflections with $I > 3\sigma(I)$
R_{int} = 0.047
θ_{max} = 27.5°, θ_{min} = 3.7°
h = −7→7
k = −15→15
l = −19→19

Refinement

Refinement on F
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.039
S = 1.04
3272 reflections
101 parameters
36 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
W = [*w*eight]*[1-(delta*F*/6*sigma*F*)^2]^2
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}
Absolute structure: Flack (1983), 925 Friedal pairs
Flack parameter: −0.03 (4)

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.93848 (10)	0.80448 (4)	0.27027 (3)	0.0686 (2)
C1	0.9383 (7)	0.9098 (3)	0.1559 (2)	0.0429 (11)
C2	0.7711 (8)	0.9981 (4)	0.1533 (3)	0.0473 (16)
C3	0.7637 (8)	1.0638 (3)	0.0755 (3)	0.0483 (16)
C4	0.9270 (10)	1.0376 (4)	0.0091 (3)	0.0507 (16)
C5	1.0976 (8)	0.9513 (4)	0.0123 (2)	0.0460 (14)
C6	1.1033 (7)	0.8838 (3)	0.0887 (3)	0.0423 (14)
C7	0.5997 (12)	1.0208 (5)	0.2295 (4)	0.071 (2)
C8	0.5866 (12)	1.1574 (5)	0.0651 (4)	0.067 (2)
C9	1.2673 (11)	0.9310 (5)	−0.0622 (4)	0.064 (2)
C10	1.2821 (11)	0.7894 (5)	0.0980 (3)	0.0597 (19)
H41	0.92110	1.08070	−0.04290	0.0610*

H71	0.69240	1.03120	0.28260	0.1071*
H72	0.50940	1.08740	0.21780	0.1072*
H73	0.49160	0.95840	0.23690	0.1071*
H81	0.62710	1.21740	0.10510	0.1008*
H82	0.59650	1.18410	0.00600	0.1008*
H83	0.42310	1.13100	0.07630	0.1010*
H91	1.24630	0.98940	−0.10560	0.0971*
H92	1.43080	0.93360	−0.04010	0.0970*
H93	1.23570	0.85840	−0.08810	0.0971*
H101	1.38670	0.80080	0.14830	0.0892*
H102	1.38000	0.78630	0.04650	0.0892*
H103	1.19650	0.71900	0.10410	0.0891*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0831 (3)	0.0727 (3)	0.0500 (2)	−0.0082 (2)	−0.0036 (2)	0.0120 (2)
C1	0.046 (2)	0.044 (2)	0.0387 (19)	−0.006 (2)	−0.004 (2)	0.0006 (17)
C2	0.047 (3)	0.044 (2)	0.051 (3)	−0.005 (2)	0.000 (2)	−0.009 (2)
C3	0.046 (3)	0.040 (2)	0.059 (3)	−0.004 (2)	−0.011 (2)	−0.007 (2)
C4	0.055 (3)	0.045 (2)	0.052 (3)	−0.007 (3)	−0.011 (3)	0.0031 (19)
C5	0.047 (3)	0.048 (2)	0.043 (2)	−0.010 (2)	−0.001 (2)	−0.0036 (18)
C6	0.041 (3)	0.041 (2)	0.045 (2)	−0.0034 (18)	−0.0062 (18)	−0.0043 (17)
C7	0.070 (4)	0.071 (3)	0.072 (4)	0.000 (3)	0.013 (4)	−0.021 (3)
C8	0.063 (4)	0.052 (3)	0.087 (4)	0.008 (3)	−0.019 (4)	−0.005 (3)
C9	0.060 (4)	0.077 (4)	0.056 (3)	−0.009 (3)	0.005 (3)	−0.011 (3)
C10	0.056 (3)	0.053 (3)	0.070 (4)	0.011 (3)	−0.006 (3)	−0.008 (3)

Geometric parameters (Å, º)

I1—C1	2.139 (3)	C7—H71	0.9600
C1—C2	1.397 (6)	C7—H72	0.9500
C1—C6	1.401 (5)	C7—H73	0.9600
C2—C3	1.416 (6)	C8—H81	0.9600
C2—C7	1.517 (8)	C8—H82	0.9500
C3—C4	1.386 (7)	C8—H83	0.9700
C3—C8	1.488 (7)	C9—H91	0.9600
C4—C5	1.392 (7)	C9—H92	0.9600
C5—C6	1.410 (6)	C9—H93	0.9600
C5—C9	1.487 (7)	C10—H101	0.9700
C6—C10	1.500 (7)	C10—H102	0.9500
C4—H41	0.9400	C10—H103	0.9600
I1—C1—C2	117.6 (3)	H71—C7—H72	109.00
I1—C1—C6	117.5 (3)	H71—C7—H73	109.00
C2—C1—C6	125.0 (3)	H72—C7—H73	110.00
C1—C2—C3	117.2 (4)	C3—C8—H81	110.00
C1—C2—C7	121.5 (4)	C3—C8—H82	108.00
C3—C2—C7	121.3 (4)	C3—C8—H83	110.00
C2—C3—C4	117.6 (4)	H81—C8—H82	109.00

C2—C3—C8	121.3 (4)	H81—C8—H83	110.00
C4—C3—C8	121.1 (4)	H82—C8—H83	109.00
C3—C4—C5	125.4 (4)	C5—C9—H91	109.00
C4—C5—C6	117.6 (4)	C5—C9—H92	109.00
C4—C5—C9	121.2 (4)	C5—C9—H93	110.00
C6—C5—C9	121.2 (4)	H91—C9—H92	109.00
C1—C6—C5	117.3 (3)	H91—C9—H93	110.00
C1—C6—C10	121.5 (4)	H92—C9—H93	110.00
C5—C6—C10	121.2 (4)	C6—C10—H101	111.00
C3—C4—H41	118.00	C6—C10—H102	109.00
C5—C4—H41	117.00	C6—C10—H103	110.00
C2—C7—H71	109.00	H101—C10—H102	108.00
C2—C7—H72	109.00	H101—C10—H103	110.00
C2—C7—H73	110.00	H102—C10—H103	109.00

I1—C1—C2—C3	−176.8 (3)	C7—C2—C3—C4	179.9 (5)
I1—C1—C2—C7	1.5 (6)	C7—C2—C3—C8	−0.3 (7)
C6—C1—C2—C3	2.1 (6)	C2—C3—C4—C5	0.4 (7)
C6—C1—C2—C7	−179.6 (4)	C8—C3—C4—C5	−179.4 (5)
I1—C1—C6—C5	178.1 (3)	C3—C4—C5—C6	0.9 (7)
I1—C1—C6—C10	−3.0 (5)	C3—C4—C5—C9	−179.2 (5)
C2—C1—C6—C5	−0.8 (6)	C4—C5—C6—C1	−0.7 (6)
C2—C1—C6—C10	178.1 (4)	C4—C5—C6—C10	−179.6 (4)
C1—C2—C3—C4	−1.8 (6)	C9—C5—C6—C1	179.4 (4)
C1—C2—C3—C8	178.0 (4)	C9—C5—C6—C10	0.4 (7)